Behavior of M[Al₂Me₆N₃] (M = K, Rb, Cs) with Aromatic Solvents and the Crystal Structures of Cs[Al₂Me₆N₃] · 2 *p*-xylene and [K · dibenzo-18-crown-6][Al₂Me₆N₃] · 1.5(1-methylnaphthalene)

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Abstract. The title compounds were synthesized by the addition of AlMe₃ to the corresponding azide suspended in an aromatic solvent. Both products were obtained as air-sensitive colorless crystals. Cs[Al₂Me₆N₃] · 2 *p*-xylene crystallizes in the monoclinic space group $C2/m$ with $a = 19.143(6)$, $b = 16.227(6)$, $c = 10.392(5)$ Å. $\beta = 114.06(2)°$, and $\rho_{calc} = 1.20$ g cm⁻³ for $Z = 4$. Refinement led to a conventional R value of 0.037 for 2179 observed reflections. The cesium atom resides on a mirror plane, and the anion is disordered about a twofold axis. The *p*-xylene molecules sandwich the cesium ion.

[K · dibenzo-18-crown-6] [Al₂Me₆N₃] · 1.5(1-methylnaphthalene) crystallizes in the monoclinic space group $P2_1/c$ with $a = 14.176(5)$, $b = 13.021(5)$, $c = 25.324(8)$ Å, $\beta = 98.23(4)°$, and $\rho_{calc} = 1.08$ g cm⁻³ for $Z = 4$. The final R value was 0.132 for 1402 observed reflections. One of the 1-methylnaphthalene molecules is disordered about a center of inversion and interacts with the potassium ion. The other solvent molecule is found roughly in layers in the lattice and also exhibits disorder of the methyl substituent. For both title compounds the AlMe₃ groups of the anion exhibit a staggered (C_s) conformation.

Key words: Aromatic···cation interactions, crown ethers, liquid clathrate, trimethylaluminum, crystal structure.

1. Introduction

It has been previously shown that equilibrium (1) is operative for M[Al₂Me₆N₃] in aromatic solvents [1].

$$M[Al_2Me_6N_3] \cdot n \text{ aromatic} \rightleftarrows M[AlMe_3N_3] + AlMe_3 + n \text{ aromatic} \qquad (1)$$
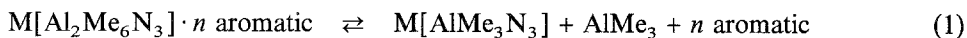
For M = K and aromatic = benzene or toluene (1) lies completely to the left. K[AlMe₃N₃] has never been obtained from these solvents. On the other hand, for M = Rb or Cs and aromatic = toluene, M[AlMe₃N₃] slowly crystallizes from solution. Since an understanding of (1) for the general M[Al₂Me₆X] is necessary with regard to the application of these substances to separations problems [2–4], we sought to extend the range of aromatics to

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include the xylenes and 1-methylnaphthalene. In the course of these studies we encountered two crystalline substances of high aromatic content. Herein we present the surprising result of the X-ray crystallographic investigations and show how they relate to the solution behavior.

2. Results and Discussion

Since for $Cs[Al_2Me_6N_3] \cdot 8.2$ toluene the equilibrium (1) is displaced slowly to the right giving crystalline $Cs[AlMe_3N_3]$, it was a surprise to find the liquid clathrate $Cs[Al_2Me_6N_3] \cdot 6$ o-xylene stable with respect to (1). (Note that all aluminum alkyl complexes are H_2O- and O_2-sensitive.) The same was found for m-xylene, and the results are summarized in Table I.

Table I. Characteristics for $Cs[Al_2Me_6N_3]$ in o-, m-, and p-xylene

Solvent	Temp $°C^a$	A/A^b
o-xylene	23	6(1)
m-xylene	23	4(1)
p-xylene	58	–

^{a} Temperature at which liquid clathrate characteristics are observed.
^{b} Maximum aromatic-to-anion ratio.

For p-xylene, a colorless crystalline solid was obtained in quantitative yield, but a single crystal X-ray diffraction analysis showed the composition to be $Cs[Al_2Me_6N_3] \cdot 2$ p-xylene. This material melts at 42–43°C, and the crystals crumble as they give up p-xylene even at room temperature in the absence of an excess of p-xylene. The liquid clathrates based on o- or m-xylene do not yield crystalline material even at $-30°C$. The results of the X-ray study provide the explanation for this substantial difference among the xylenes.

The cation environment for $Cs[Al_2Me_6N_3] \cdot 2p$-xylene is presented as Figure 1, and a stereoview of the unit cell packing is given in Figure 2. The most striking feature is that the p-xylene entities are not simply placed as molecules of crystallization in the lattice; they sandwich the cesium ion. A crystallographic mirror plane bisects each of the solvent molecules and contains the cesium ion. The $Cs \cdots C(p$-xylene) contacts range from 3.561(8) to 3.696(7) Å and average 3.64 Å (Table II). The arrangement bears a resemblance to that found in $Cs[AlMe_2I_2] \cdot p$-xylene [5]. There, the crystal was composed of layers of p-xylene molecules alternating with layers of cations and anions. The local arrangement at the cesium atom is, however, a sandwich one. The rings are eclipsed and the $Cs \cdots C(p$-xylene) average separation is 3.84 Å. In the title compound the rings are staggered, and this may be the reason for the closer $Cs \cdots C$ approach (or it may be related to the difference in overall unit cell packing). Presumably, the other xylenes because of lower inherent molecular symmetry cannot pack in either of these favorable arrays and are therefore not found as crystalline solids.

The role of the aromatic molecules in $[K \cdot DB-18-C-6][Al_2Me_6N_3] \cdot 1.5(1$-methylnaphthalene)★ is interesting, but completely different from that in $Cs[Al_2Me_6N_3] \cdot 2p$-xylene. There are two crystallographically independent 1-methylnaphthalene molecules. One is disordered about a center of inversion and is located at $z \simeq 0$ and $\frac{1}{2}$ in Figure 3. This molecule

★ DB-18-C-6 is used here to represent dibenzo-18-crown-6.
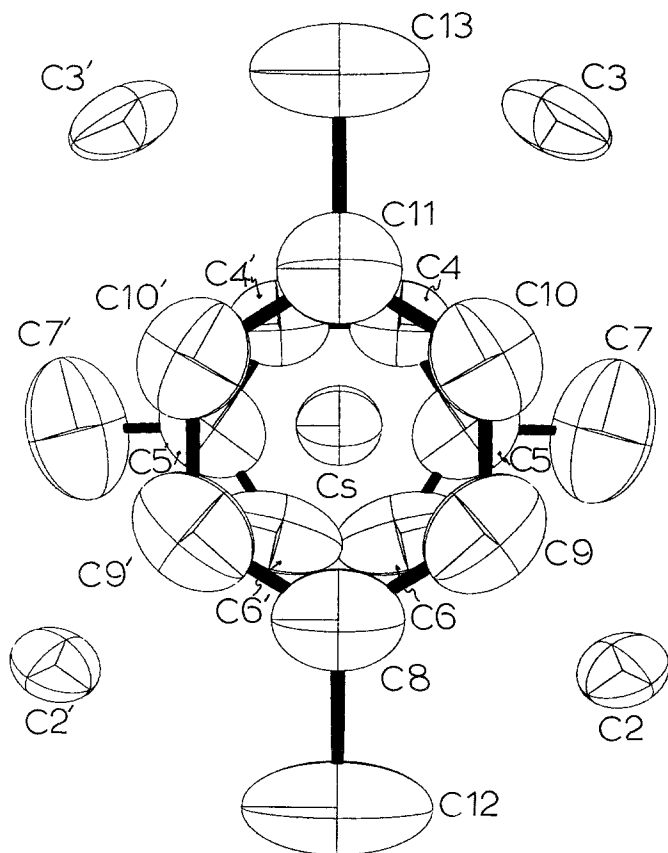
Fig. 1. Cesium ion environment in Cs[Al$_2$Me$_6$N$_3$] · 2 p-xylene. A crystallographic mirror plane contains Cs, C(8), C(11), C(12), and C(13). The C(2)- and C(3)-type atoms are methyl carbon atoms from four different anions, and the average Cs···C(methyl) separation is 3.58(4) Å.
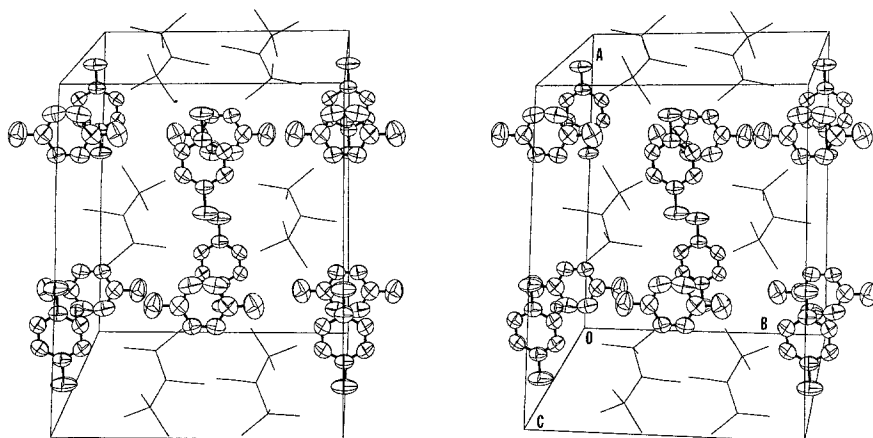

Fig. 2. Stereoscopic view of the unit cell packing in Cs[Al$_2$Me$_6$N$_3$] · 2 p-xylene.

Table II. Interatomic distances (Å) and angles (deg) for $Cs[Al_2Me_6N_3] \cdot 2\,p$-xylene

Atoms	Distance	Atoms	Distance
Al—N(1)	1.98(1)	Al′—N(1)′	1.98(1)
Al—C(1)	2.01(1)	Al′—C(1)′	2.01(1)
Al—C(2)	2.00(1)	Al′—C(2)′	2.00(1)
Al—C(3)	2.02(1)	Al′—C(3)′	1.98(1)
N(1)—N(2)	1.28(1)	N(2)—N(3)	1.13(1)
Cs···C(3)[c]	3.55(1)	Cs···C(2)	3.60(1)
Cs···C(3)′[c]	3.52(1)	Cs···C(2)′	3.63(1)
Cs···C(4)	3.685(6)	Cs···C(8)	3.561(8)
Cs···C(5)	3.696(7)	Cs···C(9)	3.588(6)
Cs···C(6)	3.664(7)	Cs···C(10)	3.616(6)
Cs···Cent1[d]	3.41	Cs···C(11)	3.662(8)
Cs···Cent2	3.34		
C(4)—C(4)[a]	1.36(2)	C(8)—C(9)	1.37(1)
C(4)—C(5)	1.38(1)	C(9)—C(10)	1.39(1)
C(5)—C(6)	1.38(1)	C(10)—C(11)	1.37(1)
C(6)—C(6)[a]	1.38(2)	C(11)—C(13)	1.53(1)
C(5)—C(7)	1.52(1)	C(8)—C(12)	1.53(1)

Atoms	Angle	Atoms	Angle
N(1)—Al—C(1)	103.8(6)	N(1)′—Al′—C(1)′	105.1(7)
N(1)—Al—C(2)	106.5(5)	N(1)′—Al′—C(2)′	105.0(5)
N(1)—Al—C(3)	96.4(6)	N(1)′—Al′—C(3)′	108.6(6)
C(1)—Al—C(2)	110.8(7)	C(1)′—Al′—C(2)′	116.6(6)
C(1)—Al—C(3)	111.5(6)	C(1)′—Al′—C(3)′	119.8(6)
C(2)—Al—C(3)	124.6(5)	C(2)′—Al′—C(3)′	100.7(5)
Al—N(1)—Al′[b]	132.5(9)	Al′—N(1)′—Al[b]	131.4(9)
Al—N(1)—N(2)	111.1(9)	Al′—N(1)′—N(2)′	113.3(9)
N(1)—N(2)—N(3)	177(2)	N(1)′—N(2)′—N(3)′	177(2)
C(4)[a]—C(4)—C(5)	122.5(5)	C(9)[a]—C(8)—C(9)	118.0(9)
C(4)—C(5)—C(6)	115.6(9)	C(9)—C(8)—C(13)	121.0(4)
C(4)—C(5)—C(7)	123.4(10)	C(8)—C(9)—C(10)	120.7(7)
C(6)—C(5)—C(7)	121.0(9)	C(9)—C(10)—C(11)	121.8(7)
C(5)—C(6)—C(6)[a]	122.0(6)	C(10)—C(11)—C(10)[a]	116.9(9)
Cent1—Cs—Cent2	176.7	C(10)—C(11)—C(12)	121.5(4)

[a] Atoms related to those in the list by x, $1 - y$, z.
[b] Atoms related to those in the list by $1 - x$, y, $1 - z$.
[c] Atoms related to those in the list by $3/2 - x$, $3/2 - y$, $1 - z$.
[d] Cent1 is the centoid of the ring conprised of atom C(4), C(4)[a], C(15), C(15)[a], C(6), C(16)[a].
Cent2, C(8), C(9), C(9)[a], C(10), C(10)[a], C(11).

appears to interact weakly with the potassium ion in the crown and roughly fills the basket in the crown unit (Figure 4). The other aromatic packs in layers at roughly $z = \frac{1}{4}$ and $\frac{3}{4}$. Thus, the lattice contains aromatic molecules with cation···aromatic interactions of Types **1** and **3** in [6].

The anion is also of interest. One can envisage two arrangements of the aluminum alkyl units attached to the azide ion, one eclipsed (C_{2v} point symmetry) and one staggered (C_s point symmetry). In the structure of $K[Al_2Me_6N_3]$ there are two crystallographically independent
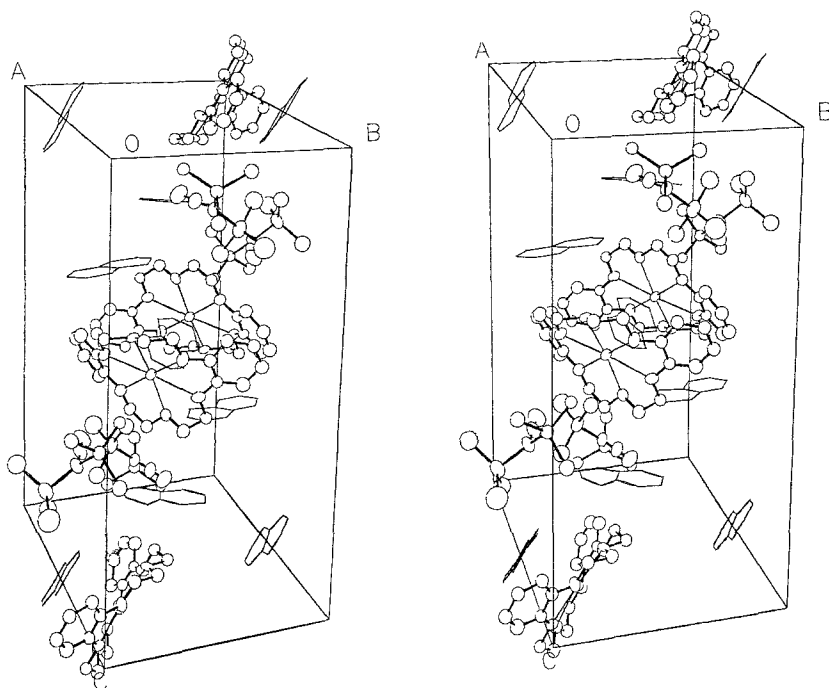
Fig. 3. Stereoscopic view of the unit cell packing in [K·dibenzo-18-crown-6][Al₂Me₆N₃]·1.5(1-methyl-naphthalene). The methyl groups of the solvent molecules were so badly disordered they could not be located, and therefore they are not shown.
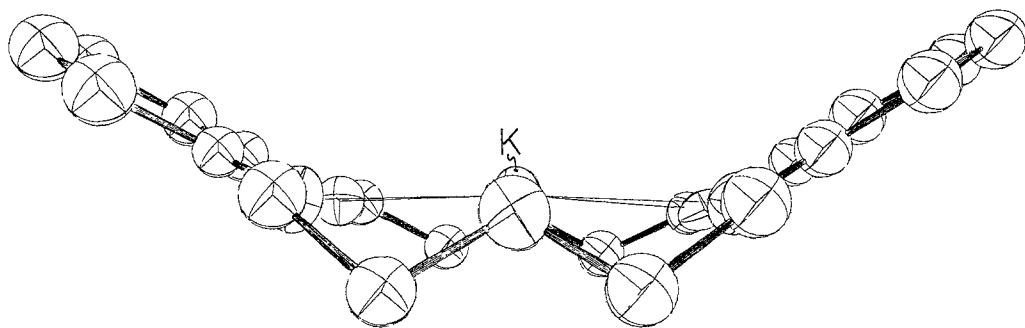
Fig. 4. [K·dibenzo-18-crown-6]⁺ cation showing the basket-like configuration of the crown ether moiety.

anions, one of each possible symmetry [7]. However, in the title compounds both anions exhibit the staggered configuration. Figure 5 shows the disordered situation for the cesium complex (discussed in detail in the Experimental), while Figure 6 shows the anion in the potassium complex. Bond lengths and angles in the anions (Tables II and III) compare favorably with each other and with those in K[Al₂Me₆N₃] [7].
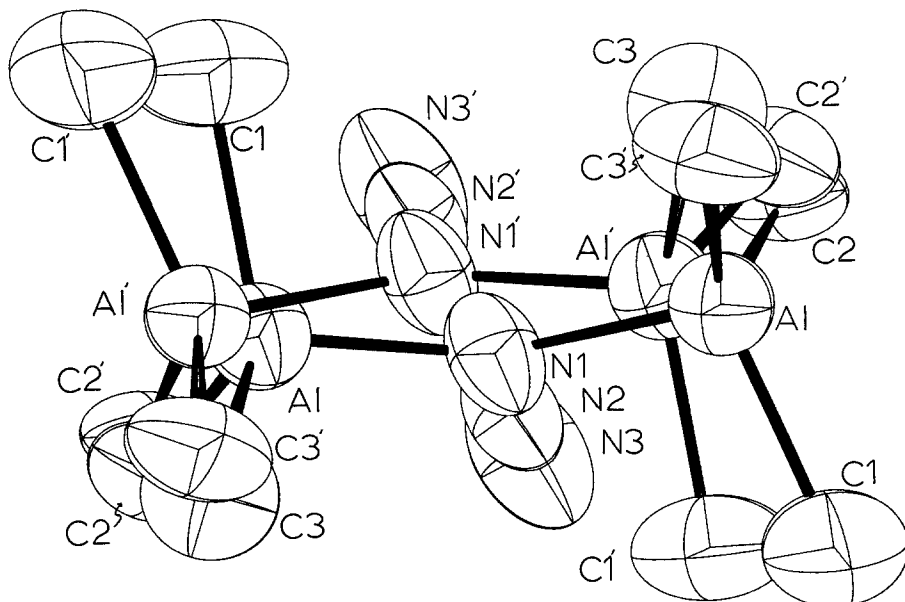
Fig. 5.   Anion of $Cs[Al_2Me_6N_3] \cdot 2\,p$-xylene illustrating the crystallographic disorder about the two-fold axis.
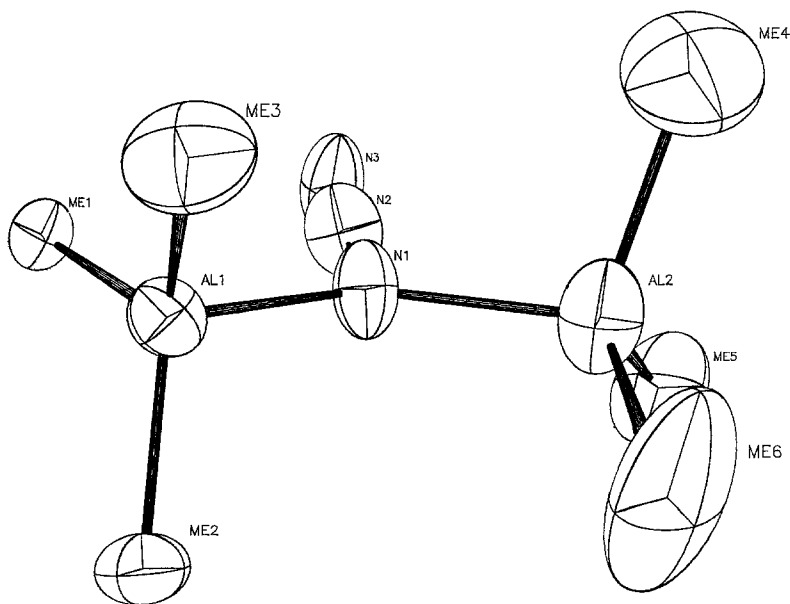
Fig. 6.   Anion of $[K \cdot dibenzo$-18-$crown$-$6]\,[Al_2Me_6N_3] \cdot 1.5(1$-methylnaphthalene$)$.

3. Experimental

All manipulations were carried out in an inert atmosphere glove box. Solvents were dried in the usual manner. Cesium azide, potassium azide, and trimethylaluminum were obtained from Alfa Ventron Corporation and used without further purification.

Table III. Bond distances (Å) and angles (deg) for the anion of [K·dibenzo-18-crown-6] [Al₂Me₆N₃] · 1.5(1-methylnaphthalene)

Atoms	Distance	Atoms	Distance
Al(1)—N(1)	2.06(3)	Al(1)—Me(1)	2.00(3)
Al(1)—Me(2)	2.02(3)	Al(1)—Me(3)	1.99(4)
Al(2)—N(1)	2.00(3)	Al(2)—Me(4)	2.03(5)
Al(2)—Me(5)	1.97(4)	Al(2)—Me(6)	1.99(4)
N(1)—N(2)	1.22(4)	N(2)—N(3)	1.11(4)

Atoms	Angle	Atoms	Angle
N(1)—Al(1)—Me(1)	102(1)	N(1)—Al(1)—Me(2)	106(1)
Me(1)—Al(1)—Me(2)	116(1)	N(1)—Al(1)—Me(3)	103(2)
Me(1)—Al(1)—Me(3)	114(2)	Me(2)—Al(1)—Me(3)	114(2)
N(1)—Al(2)—Me(4)	100(2)	N(1)—Al(2)—Me(5)	109(2)
Me(4)—Al(2)—Me(5)	114(2)	N(1)—Al(2)—Me(6)	107(2)
Me(4)—Al(2)—Me(6)	111(3)	Me(5)—Al(2)—Me(6)	115(3)
Al(1)—N(1)—Al(2)	128(2)	Al(1)—N(1)—N(2)	114(3)
Al(2)—N(1)—N(2)	117(3)	N(1)—N(2)—N(3)	176(5)

3.1. SYNTHESIS OF Cs[Al₂Me₆N₃] · 2 p-xylene

Cesium azide (1.75 g, 10 mmol) was mixed with 25 ml of p-xylene in a Fisher–Porter tube. Trimethylaluminum (2 ml, 20 mmol) was syringed into the mixture which was then sealed and heated at 60°C for 5 h in an oil bath. Slow cooling to room temperature produced the title compound as large transparent highly air-sensitive crystals. The melting point of Cs[Al₂Me₆N₃] · 2 p-xylene was determined using a Thomas Hoover melting point apparatus and found to be 42–43°C. Above 58°C, normal liquid clathrate behavior was observed.

The reaction described above was also carried out in o- and m-xylene. The A/A numbers were obtained from the integration of NMR spectra recorded on a Varian EM-360 spectrometer.

3.2. SYNTHESIS OF [K · DB-18-C-6] [Al₂Me₆N₃] · 1.5(1-methylnaphthalene)

A slurry of potassium azide (0.404 g, 5.0 mmol) and DB-18-C-6 (1.80 g, 5.0 mmol) was prepared in 25 ml of 1-methylnaphthalene in a screw-top tube. Trimethylaluminum (1.0 ml, 10.0 mmol) was syringed into the mixture and the tube was sealed. A homogeneous solution resulted after heating for 12 hr at 80°C. No evidence of a liquid clathrate was noted. After standing at room temperature for two weeks, the solution afforded large, colorless, air-sensitive crystals of the title compound.

3.3. X-RAY DATA COLLECTION, STRUCTURE DETERMINATION, AND REFINEMENT FOR Cs[Al₂Me₆N₃] · 2 p-xylene

Single crystals of the air-sensitive compound were sealed under N₂ in thin-walled glass capillaries. Final lattice parameters as determined from a least-squares refinement of $((\sin \theta)/\lambda)^2$ values for 15 reflections ($\theta > 20°$) accurately centered on the diffractometer are given in Table IV. The space group was found to be C2, Cm or C2/m. The centrosymmetric C2/m was initially chosen and was shown to be correct by successful refinement.

Table IV.   Crystal data and summary of intensity data collection and structure refinement

Compound	$Cs[Al_2Me_6N_3] \cdot 2\,p$-xylene	[K · dibenzo-18-crown-6] [Al$_2$Me$_6$N$_3$]-1.5(1-methylnaphthalene)
Mol. wt.	531.4	750.7
Space group	$C2/m$	$P2_1/c$
Cell constants		
a, Å	19.143(6)	14.176(5)
b, Å	16.227(6)	13.021(5)
c, Å	10.392(5)	25.324(8)
β, deg	114.06(2)	98.23(4)
Cell vol., Å^3	2947.6	4626.3
Molecules/unit cell	4	4
ρ(calc), g cm^{-3}	1.20	1.15
μ(calc), cm^{-1}	13.52	1.5
Radiation	Mo$K\alpha$	Mo$K\alpha$
Max. crystal dimensions, mm	$0.30 \times 0.20 \times 0.40$	$0.45 \times 0.31 \times 0.30$
Scan width	$0.80 + 0.20 \tan\theta$	$0.80 + 0.20 \tan\theta$
Standard reflections	400, 060	400, 060, 0012
Reflections measured	2960	3705
2θ range	2–50°	0–40°
Reflections observed	2179	1502
No. of parameters varied	184	255
GOF	1.07	1.34
R	0.037	0.148
R_w	0.044	0.153

Data were collected on an Enraf-Nonius CAD-4 diffractometer by the θ-2θ scan technique. The method has been previously described [8]. A summary of data collection parameters is given in Table IV. The intensities were corrected for Lorentz and polarization effects, but not for absorption.

Calculations were carried out with the XFLS program [9]. Neutral atom scattering factors for Cs, Al, N, and C were taken from Cromer and Waber [10], and the scattering for cesium was corrected for the real and imaginary components of anomalous dispersion using the table of Cromer and Liberman [1]. Scattering factors for H were from [12].

The position of the cesium atom on a mirror plane was revealed by the inspection of a Patterson map. A difference Fourier map phased on the cesium atom readily revealed the positions of the nonhydrogen atoms but indicated some type of disorder of the anion about a two-fold axis. Refinement in the noncentric space group Cm produced a higher R factor and still showed evidence of disorder in the anion. Refinement in $C2$ produced high correlations between atoms that would have been related by the mirror in $C2/m$. For these reasons $C2/m$ was taken as the correct choice and the disorder solved. Two peaks of the same intensity were found on one side of the two-fold axis and corresponded to two Al atoms occupying each site 50% of the time. The three nitrogen atoms were slightly off the two-fold axis with again equal occupancy on each side. The fractionally disordered carbons were found last. All atoms in the disordered anion were given occupancy factors of 0.5. These values were not refined. Least-squares refinement with isotropic thermal parameters led to $R = \Sigma \|F_o| - |F_c\|/\Sigma|F_o| = 0.063$. The hydrogen atoms of the p-xylene rings that could be placed in calculated positions were fixed 0.98 Å from the bonded carbon atom and were not

refined. No other hydrogen atoms were located. Refinement of the nonhydrogen atoms with anisotropic temperature factors led to final values of $R = 0.037$ and $R_w = 0.044$. A final difference Fourier showed no feature greater than 0.5 e$^-$/Å^3. The weighting scheme was based on unit weights; no systematic variation of $w(|F_o| - |F_c|)$ vs. $|F_o|$ or $(\sin\theta)/\lambda$ was noted. The final values of the positional parameters are given in Table V [13].

Table V.   Final fractional coordinates for Cs[Al$_2$Me$_6$N$_3$] · 2 p-xylene

Atom	x/a	y/b	z/c
Cs	0.7188(1)	0.5000(0)	0.5352(1)
Al[a]	0.5487(2)	0.7760(2)	0.3601(4)
Al'	0.5461(2)	0.7885(2)	0.4025(4)
N(1)	0.4893(10)	0 8285(6)	0.4547(11)
N(2)	0.4652(6)	0.8996(8)	0.4025(13)
N(3)	0.4469(8)	0.9630(8)	0.3562(19)
C(1)	0.4715(9)	0.7596(12)	0.1610(15)
C(1)'	0.4510(8)	0.7799(11)	0.2230(15)
C(2)	0.5848(7)	0.6679(8)	0.4559(14)
C(2)'	0.6013(6)	0.6832(7)	0.4813(12)
C(3)	0.6172(7)	0.8733(9)	0.3754(13)
C(3)'	0.6322(7)	0.8587(8)	0.4092(14)
C(4)	0.7519(4)	0.4582(5)	0.2170(6)
C(5)	0.6865(6)	0.4127(6)	0.1880(7)
C(6)	0.6208(4)	0.4575(6)	0.1580(7)
C(7)	0.6848(8)	0.3191(7)	0.1838(11)
C(8)	0.6595(5)	0.5000(0)	0.8195(9)
C(9)	0.6998(4)	0.5724(4)	0.8466(6)
C(10)	0.7792(4)	0.5720(4)	0.8948(6)
C(11)	0.8201(5)	0.5000(0)	0.9206(9)
C(12)	0.5722(6)	0.5000(0)	0.7673(12)
C(13)	0.9074(5)	0.5000(0)	0.9728(12)
H(1)[C(4)]	0.797	0.431	0.235
H(2)[C(6)]	0.580	0.429	0.138
H(3)[C(9)]	0.676	0.627	0.837
H(4)[C(10)]	0.808	0.628	0.916

[a] Occupancy factors for the atoms of the disordered anion are 0.5.

3.4. X-RAY DATA COLLECTION, STRUCTURE DETERMINATION, AND REFINEMENT FOR [K · DB-18-C-6] [Al$_2$Me$_6$N$_3$] · 1.5(1-methylnaphthalene)

Data was collected as described in Section 3.3. The associated parameters are given in Table IV. The structure was solved by application of MULTAN [14], and refinement of the parameters of the cation and anion proceeded smoothly using SHELX [15]. The two crystallographically independent solvent molecules were located by employing a series of difference Fourier maps, but the one found about the center of inversion exhibits disordered methyl groups. The second 1-methylnaphthalene molecule is also disordered, but in an apparently more random fashion. (It is worth noting that the scattering in high aromatic content crystals such as this one is greatly reduced because of the high thermal motion which invariably occurs.) The lack of observed reflection data did not permit a full anisotropic refinement; the anion, the potassium atom, and the oxygen atoms of the crown ether were

refined with anisotropic thermal parameters, but the carbon atoms of the crown ether were given only isotropic parameters. The solvent molecules were located and then converted to idealized naphthalene units and refined as rigid groups. Initially, the isotropic B of each carbon atom of the 1-methylnaphthalene molecule was allowed to vary. Later, the B's were fixed and only the group (x, y, z)'s and rotation angles were refined. This procedure led to $R = 0.132$. No unaccounted electron density was noted on a final difference Fourier map. The final values of the positional parameters are given in Table VI [13].

Table VI. Final fractional coordinates for [K·dibenzo-18-crown-6] [$Al_2Me_6N_3$]·1.5(1-methylnaphthalene)

Atom	x/a	y/b	z/c	U_{eqv}
K(1)	0.2945(5)	0.3128(6)	0.5014(3)	0.081
O(1)	0.414(1)	0.154(2)	0.4837(8)	0.088
O(2)	0.252(2)	0.208(2)	0.4064(7)	0.085
O(3)	0 136(1)	0.371(2)	0.4330(9)	0.095
O(4)	0.162(1)	0.460(2)	0.5239(9)	0.089
O(5)	0.317(1)	0.396(2)	0.5992(7)	0.075
O(6)	0.446(1)	0.246(2)	0.5741(8)	0.087
C(1)	0.393(2)	0.111(3)	0.431(1)	0.087
C(2)	0.288(2)	0.104(3)	0.413(1)	0.083
C(3)	0.154(2)	0.210(3)	0.388(1)	0.102
C(4)	0.126(2)	0.320(3)	0.383(1)	0.087
C(5)	0.106(2)	0.472(3)	0.432(2)	0.092
C(6)	0.071(2)	0.525(3)	0.384(1)	0.097
C(7)	0.045(2)	0.628(3)	0.394(1)	0.101
C(8)	0.057(2)	0.672(3)	0.443(1)	0.099
C(9)	0.094(3)	0.619(3)	0.487(1)	0.106
C(10)	0.118(2)	0.515(3)	0.482(1)	0.078
C(11)	0.183(2)	0.504(3)	0.575(1)	0.088
C(12)	0.232(3)	0.431(3)	0.614(1)	0.102
C(13)	0.365(2)	0.333(3)	0.637(1)	0.092
C(14)	0.459(2)	0.298(3)	0.623(1)	0.088
C(15)	0.524(2)	0.201(3)	0.552(1)	0.084
C(16)	0.617(3)	0.212(3)	0.584(1)	0.107
C(17)	0.688(3)	0.168(3)	0.556(2)	0.127
C(18)	0.668(3)	0.125(3)	0.510(2)	0.126
C(19)	0.585(3)	0.114(3)	0.480(2)	0.123
C(20)	0.507(3)	0.157(2)	0.504(1)	0.076
Al(1)	0.1158(7)	0.4857(9)	0.0945(4)	0.092
Al(2)	0.2328(9)	0.658(1)	0.1977(6)	0.130
N(1)	0.227(2)	0.535(2)	0.150(1)	0.100
N(2)	0.303(3)	0.491(3)	0.147(2)	0.118
N(3)	0.373(2)	0.455(4)	0.147(2)	0.159
Me(1)	0.176(2)	0.374(2)	0.057(1)	0.085
Me(2)	0.012(2)	0.442(3)	0.136(1)	0.119
Me(3)	0.084(2)	0.611(3)	0.051(1)	0.122
Me(4)	0.299(4)	0.759(4)	0.154(2)	0.200
Me(5)	0.307(3)	0.623(4)	0.267(2)	0.182
Me(6)	0.099(3)	0.701(6)	0.200(3)	0.342
A(0)[a]	0 531(1)	0.464(3)	0.486(1)	0.123
A(1)	0.488(1)	0.424(3)	0.435(1)	0.155
A(2)	0.407(1)	0.447(3)	0.414(1)	0.150
A(3)	0.352(1)	0.512(3)	0.425(1)	0.145

Table VI   (continued)

Atom	x/a	y/b	z/c	U_{eqv}
A(4)	0.378(1)	0.559(3)	0.484(1)	0.126
Z(1)[b]	0.822(2)	0.733(2)	0.255(1)	0.227
Z(2)	0.737(2)	0.796(2)	0.247(1)	0.204
Z(3)	0.742(2)	0.781(2)	0.229(1)	0.189
Z(4)	0.586(2)	0.689(2)	0.212(1)	0.186
Z(5)	0.652(2)	0.610(2)	0.219(1)	0.180
Z(6)	0.638(2)	0.505(2)	0.211(1)	0.208
Z(7)	0.716(2)	0.438(2)	0.219(1)	0.221
Z(8)	0.807(2)	0.476(2)	0.235(1)	0.187
Z(9)	0.821(2)	0.582(2)	0.243(1)	0.287
Z(10)	0.743(2)	0.648(2)	0.235(1)	0.121

[a] The symbol A indicates one of the ring carbon atoms of the 1-methylnaphthalene molecule which is disordered about a center of symmetry.

[b] The symbol Z indicates one of the ring carbon atoms of the 1-methylnaphthalene molecule which resides in a general position in the unit cell. The methyl groups were not located on either of the solvent molecules.

Acknowledgement

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